Status of the Calipso program

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Introduction

The Bay area is well endowed with national facilities. There are three major DOE laboratories, Lawrence Berkeley National Laboratory (LBNL) in Berkeley, Lawrence Livermore National Laboratory (LLNL) in Livermore and the Stanford Linear Accelerator Facility (SLAC) at Stanford. We are well equipped with advanced accelerator sources with two synchrotrons (SPEAR III at SLAC and the ALS at LBNL) and a Free Electron Laser (FEL) at SLAC. There are also about twenty high-pressure groups with-in a two hour drive of the Bay Area. This makes for very fertile ground for high-pressure research and indeed Bay Area high-pressure research teams have been at the forefront of many of the developments in this field including the exploitation of synchrotron radiation. A consortium of Bay Area groups made use of the SPEAR II synchrotron at SLAC. This was done in a parasitic mode with use of a shared hutch and build up and break down of experimental equipment each beamtime period. With the upgrade to SPEAR II to give the current SPEAR III machine there was a shutdown of over a year and no guarantee of beamtime access for the high-pressure consortium after. This led the consortium to approach the ALS science advisory council with a proposal to establish dedicated high-pressure facilities at the ALS. This proposal was accepted, the high-pressure community was given a hunting license to raise funds for the development of two beamlines at the ALS and a high-pressure expert was recruited by the ALS to lead this development (which brought me to the Bay Area). Initial demonstration high-pressure experiments started in 2001-2002 with measurements on beamline 7.3.3 (x-ray) and 1.4 (infrared). In 2003 a part share in beamline 11.3 was obtained and 30% of the beamtime allocated to high-pressure. Funding was obtained for a dedicated high-pressure facility by Raymond Jeanloz and Paul Alivisatos in 2003 with $1M coming from DOE, $1M from the University of California and $1M from LBNL. Building of this beamline (12.2.2) was completed at the end of 2004 with commissioning during 2005 and first users in January 2006. A summary of the current facilities at the ALS is contained in appendix I.

Ethos and strategy

Early on in the development of these current facilities the west coast high-pressure community met and devised the ethos for west coast high-pressure developments. This is called Calipso (the CALifornia hIgh Pressure Science Observatory). The core value of Calipso is that all of our facilities should be accessible by all scientists whether or not they are high-pressure specialists. The concept being that scientists from any background should be able o come to our facilities with their sample and leave with the information that they need. We have used this principle to guide the development of beamline 12.2.2

aiming at the highest degree of automation and ease of use. To make an easy to use facility we need to focus on a subset of techniques to minimize change over between modes and user training. We decided to only work with diamond anvil cells and not to develop any energy-dispersive facilities. We developed the concept of each beamline end station as a diamond cell surrounded by a cluster of techniques that can be applied at will by the user without any need to call on the beamline scientists for assistance. Guided by these principles we built beamline 12.2.2 with a brightness preserving beamline with automated alignment to about 5 µm and two end stations: end station 1 (ES1) with a large focal spot (100x120µm) equipped with a CCD detector for resistively heated diamond anvil cell experiments and end station 2 (ES2) with a microfocus (10x10µm) for laser heated diamond anvil cell experiments. We also developed a high-pressure laboratory with all of the necessary equipment for diamond cell alignment, gasket preparation, sample loading and pressure measurement and provided a suite of diamond cells. The current status of these facilities is summarized in appendix II and a statement our development plan in appendix III.

In our view the key component of providing user friendly, highly productive facilities is a high level of automation and beamline readiness together with highly trained and motivated beamline scientists to maintain the facilities and provide user training and support.

Continuing support and resource distribution

Monies to support Calipso operations come from LBNL, LLNL and COMPRES. Currently we get each year about $80k for consumables and $100k for capital equipment from the ALS, $250k from COMPRES and $50 to $100k from LLNL. Due to charges for funds transfer and the various overhead rates we choose to use the ALS money for consumables and capital investment, the LLNL money for capital items and the COMPRES money to support staff. The current west coast team is comprised of:

Raymond Jeanloz	UC Berkeley	COMPRES grant PI. Provides guidance and oversight.
Simon Clark	ALS	Calipso program manager. Provides project management. .. Responsible for infrared systems and data analysis and interpretation and user support.
Robin Titus	ALS	Associate beamline scientist (50%). Provides mechanical design and support.
Martin Kunz	COMPRES	Beamline scientist. Responsible for x-ray systems and user support.
Sander Caldwell	COMPRES	Associate beamline scientist. Responsible for laser heating system and user support.
Jinyuan Yan	COMPRES	Post-doc. Responsible for developing automated data analysis procedures.

So basically, there are about 2.5 people providing direct user support for 1.5 beamlines compared with the DOE standard of 4 people. Since the ALS operates for about 300

days/annum that means that our staff are fully occupied with direct user support about 75% of their time, when we take into account weekends and vacation, making it hard to fit in routine administration, training and meetings let alone continuing education or research. Phasing out the use of beamline 11.3 will improve this situation but if we want to fully exploit our facilities in the manner described above we will need to increase the staff by at least 1.5 people. We have requested an extra person with a background in spectroscopy from the COMPRES II grant application and a new beamline scientist position form the ALS.

Beamtime take up and deliverables

Beamtime on beamline 12.2.2 is divided between general users and approved programs. General users get 25% of the beamtime. There are three approved programs on beamline 12.2.2 at the moment: UC, LLNL and COMPRES. These we call the high-pressure consortium. Beamtime is divided between them according to the annual support they provide and amortized capital investment. These amounts vary year by year. The actual beamtime division is contained in appendix IV.

A summary of beamtime applications is contained in appendix V, publications in appendix VI and dissertations in appendix VII.

Future developments

The current focus is on completing the development of the facilities that we have and exploiting them. We are, however, undertaking two major developments: upgrade of endstation 1 for single crystal diffraction and development of the LCLS for high-pressure shockwave measurements.

1. **ES1 upgrade**. A new thrust for the HP community is making very precise structural measurements at higher pressures. To achieve this we need to develop single crystal diffraction facilities at our synchrotron sources. A COMPRES funded workshop brought the community together to discuss this and a subsequent successful grant application as provided the money for us to upgrade ES1 with the necessary goniometry to allow single crystal measurements. The long term plan is to move the Brillouin system on to ES1 to go with the single crystal setup and eventually to add laser heating as well. Further down the line we plan to propose to develop beamline 12.2.1 for single crystal diffraction supporting both the high-pressure single crystal diffraction and the chemical crystallography program at the ALS.
2. **LCLS.** This facility will come on line in about four years from now. It will allow us to collect a whole diffraction pattern, suitable for accurate structure determination, using one 120fs x-ray pulse from materials at the high pressures and temperatures generated by shockwaves. This will allow us to greatly extend the range of pressures and temperatures that we can access for these measurements and will also overcome many of the limitations imposed on us by using diamond anvil cells. We intend to contribute to this development as part of the warm condensed matter consortium.

Appendix I. Advanced Light Source High-pressure group status October 2006

Personnel

Simon Clark	Group leader	510-495-2442	smclark@lbl.gov
Martin Kunz	Beamline Scientist responsible for x-ray experiments	510-486-6789	mkunz@lbl.gov
Sander Caldwell	Associate beamline scientist responsible for heating at high pressure	510-486-7849	wacaldwell@lbl.gov
Jinyuan Yan	Post-doc responsible for developing data analysis software	510-486-4932	jyan@lbl.gov
Robin Titus	Associate beamline scientist responsible for mechanical engineering and design	510-495-2646	rctitus@lbl.gov

Experimental facilities

1. Synchrotron facilities
    a. Beamline 12.2.2. High energy x-ray beamline equipped for monochromatic diffraction and x-ray absorption spectroscopy (being commissioned) with laser and resistively heated samples held in diamond anvil cells. Includes on-line ruby fluorescence, Raman (under development), spectroradiography, x-ray shadography and visible light imaging. Currently used with powder, liquid and amorphous samples. Being upgraded to allow high pressure-temperature single crystal diffraction. Cryostat available but as yet not commissioned.
    b. Beamline 11.3. Low energy x-ray beamline equipped for monochromatic powder diffraction from samples held in diamond anvil cells. Includes on-line imaging system.
    c. Beamline 1.4. Infrared beamline equipped with suitable diamond anvil cells and resistively heated diamond anvil cells.
2. Laboratory facilities

a. High-pressure laboratory equipped with sample preparation equipment and facilities for loading and preparing diamond anvil cells including microdrilling equipment for gasket preparation.
b. Off-line ruby fluorescence system.
c. Raman system. (ready for assembly once laser purchased)
d. Brillouin scattering system. (under commissioning)

Appendix IIa: Status of 12.2.2 HP systems: July 2006.

Status of 12.2.2 HP systems (July 2006)

	User Facility	Needs Improvement	Commissioning	Being Assembled	Parts Delivered	Parts Ordered	In design	In conceptual design	Under consideration
Diamond cells	★								
Laser heating		★							
Resistive heating							★		
Cryostat					★				
Shockwave									★
Powder diffraction (Axial)	★								
Powder diffraction (Radial)	★								
Single Xtal polychromatic			★						
Single Xtal monochromatic							★		
X-ray imaging	★								
X-ray abs. spectroscopy			★						
Ruby Fluor. (off-line)	★								
Ruby Fluor. (on-line)				★					
Raman Spectroscopy					★				
Brillouin Spectroscopy				★					
Conductivity									★
Loading microscopes	★								
Spark eroder	★								
Micro-drill						★			
Cryogen loading	★								
Gas loading									★

Appendix IIb: Status of 12.2.2 HP systems: target for July 07.

Status of 12.2.2 HP systems (Target for July 2007)

	User Facility	Needs Improvement	Commissioning	Being Assembled	Parts Delivered	Parts Ordered	In design	In conceptual design	Under consideration
Diamond cells	★								
Laser heating	★								
Resistive heating	★								
Cryostat			★						
Shockwave									★
Powder diffraction (Axial)	★								
Powder diffraction (Radial)	★								
Single Xtal polychromatic	★								
Single Xtal monochromatic			★						
X-ray imaging	★								
X-ray abs. spectroscopy	★								
Ruby Fluor. (off-line)	★								
Ruby Fluor. (on-line)	★								
Raman Spectroscopy			★						
Brillouin Spectroscopy	★								
Conductivity									★
Loading microscopes	★								
Spark eroder	★								
Micro-drill	★								
Cryogen loading	★								
Gas loading									★

Appendix III. West coast status and development plan

Our aim

The resolution of the outstanding issues surrounding the composition and evolution of the Earth and other planetary bodies using high-pressure, high-temperature mineralogy.

Our approach

Use synchrotron and free electron laser generated x-rays and synchrotron and laboratory based spectroscopies to accurately determine phase boundary equilibria for planetary systems and volume and elastic properties of planetary materials.

The methods that we use

Synchrotron based x-ray diffraction, x-ray spectroscopy, infrared and imaging of samples held at high-pressure and temperatures in diamond anvil cells with resistive and laser heating and on-line pressure determination. Supplementary laboratory based Raman, Brillouin and Impulsive Light Scattering measurements. Shockwave generated high-pressures and temperatures probed by ultra short pulses of x-rays.

Samples consist of powders or single crystals held in hydrostatic fluids.

We aim to attract the best user groups and the best experiments from around the world by providing highly automated, user friendly, experimental facilities optimized for our target user community and to vigorously pursue a program of in house research in order to both meet our primary aim and to validate our experimental systems. Also, we aim to engage and train the next generation of scientists who will continue this work until our goals are achieved.

Available and developing experimental facilities

1. **Beamline 12.2.2, Diffraction and laser heating.** This is a hard x-ray (8-35keV) focused synchrotron beamline situated on one of the superbend sources at the Advanced Light Source. It contains two end stations. End station 1 has a large x-ray focal spot (100x80μm) and is equipped for x-ray diffraction measurements from powders held in diamond anvil cells. End station 2 has a smaller focal spot (10x10μm) and is equipped with a double sided laser heating system, ruby fluorescence system and Mar345 image plate system. Both end stations are equipped with sufficient goniometry to allow automatic alignment and setting of the sample to detector distance. Endstation 2 is currently being upgraded with additional goniometry to allow high-pressure single crystal diffraction measurements.

2. **Beamline 1.4 Infrared.** This is a state of the art synchrotron infrared beamline situated on one of the bending magnets at the Advanced Light Source. It is equipped with a fully automated Thermo Nicolet Nexus 870 FTIR and having a diffraction limited focal spot. Two low profile diamond cells suitable for working with this end station are available one is equipped with a external resistive heater.
3. **Beamline 11.3.1, Diffraction.** This is a medium energy synchrotron x-ray beamline which benefits from x-rays from one of the ALS bending magnets. It is equipped to allow high-pressure diffraction measurements from samples contained in diamond anvil cells using a Bruker CCD.
4. **Beamline 12.2.1 Single crystal diffraction.** Beamline 12.2.1 is earmarked by the ALS Science Advisory Council for high-pressure research. This is a superbend beamline which delivers the hardest x-rays available at the ALS. We hope to be able to move our single crystal high-pressure diffraction equipment onto this beamline during the next two years.
5. **Beamline 5.3.1. Shockwaves and diffraction.** This beamline is optimized for the production of ultra-short x-ray pulses from a slicing source. The current femtosecond program is moving to a new undulator beamline leaving this beamline free. We are thinking of equipping beamline with a laser driven shockwave system and imaging plate x-ray detector to allow x-ray studies ahead of the shockwave beamline at LCLS coming on line.
6. **High-Pressure Laboratory.** The high-pressure laboratory contains all of the equipment necessary to align and load diamond anvil cells. This includes microscopes, spark eroder, cryogen loading, etc. The laboratory also contains two optical tables upon which we are currently assembling Brillouin and Raman systems.

Current level of support

The Calipso program manager (Simon Clark), 50% of an associate beamline scientist (Robin Titus), the beamline 1.4 and 11.3 scientists, a pool of engineers, technicians and craftsmen as well as other support personnel are supported directly by the ALS. Beamline 12.2.2 receives $60k and the high-pressure laboratory $30k from the ALS for consumables. COMPRES support two beamline scientists (Martin Kunz and Sander Caldwell), together with their travel support and some consumables, and a post-doc developing automated data analysis software (Jinyuan Yan). LLNL donates equipment each year to a value of about $150k.

Proposed future level of support

a. Short term

Our core facilities are almost completely commissioned and little capital investment is necessary over the next five years. We could greatly increase productivity on the infrared beamline by addition of an on-line ruby fluorescence system ($40k) and additional diamond cells dedicated to the beamline scientist's science programs would be useful

($40k). The major risk to delivery of the COMPRES program is in breakdown of our heating laser. We would propose to replace our current heating system with two solid state lasers ($60k). The current laser can be installed on our off-line Brillouin system and moved back into the 12.2.2 hutch at short notice if a breakdown occurs.

Two additional members of staff are needed. One specializing in spectroscopy, to give us full coverage of the expertise necessary to maintain our facilities at the cutting edge technologically and to provide much need extra user support. The other providing software support to allow us to completely integrate our beamline functionality into one graphical user interface and to fully automate the data collection and processing functions.

b. Longer term

Beamline 12.2.1 gives us the opportunity to devlelop the only dedicated high-pressure single crystal beamline in the world. This will satisfy a well know need. An investment of about $400k from COMPRES, matched by funds from ALS, is all that is necessary to make this a reality.

The developments on beamline 5.3.1 together with the shockwave beamline at the LCLS are truly unique and an area where COMPRES can show outstanding leadership by championing. The major need here is manpower to get this area going.

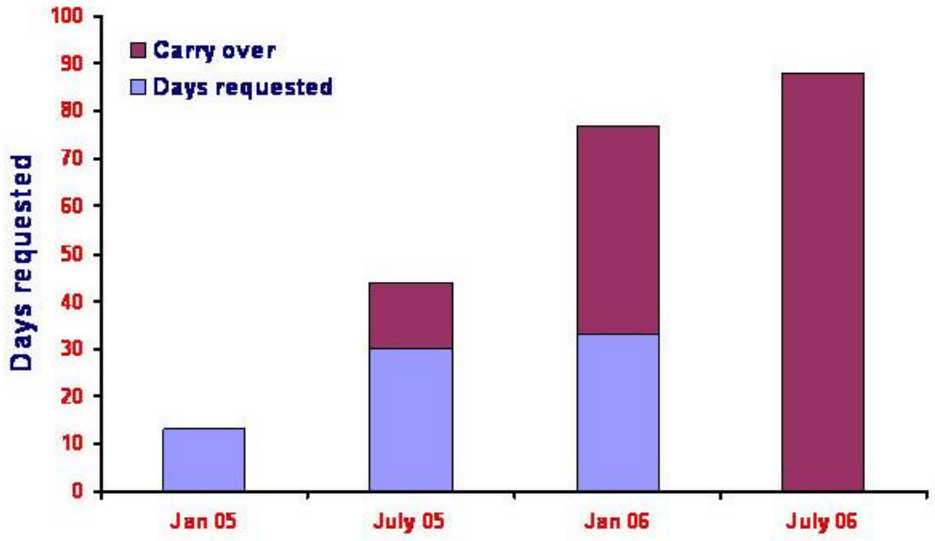
Appendix IV. Division of beamtime on beamline 12.2.2.

Consortium members and beamtime division (%)

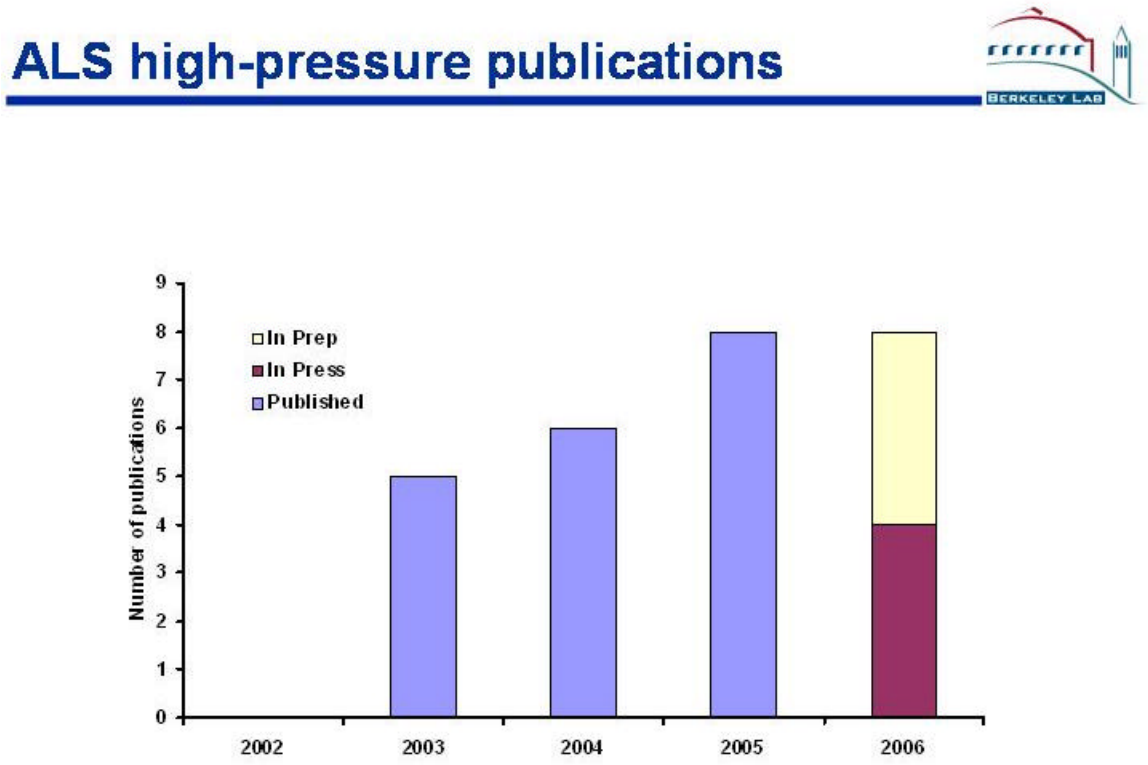
		2005	2006	2007	2008	2009
UC	Jeanloz	14	13.5	13	13	9.5
	Alivisatos	14	13.5	13	13	9.5
	Tolbert	3.5	3.5	3	3	2.5
	Williams	3.5	3.5	3	3	2.5
LLNL		15	14	14	13	10
COMPRES		23	24	26	29	35
G.U.s		0	25	25	25	25
Maintenance, Commissioning and DD.		70	20	20	20	20

Appendix V. Summary of beamtime applications

12.2.2 beamtime requests



Appendix VI. Summary of publications



Appendix VII. Summary of dissertations